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# Spectral and Transmittance Properties of Ho<sup>3+</sup> Doped in Zinc Lithium Lead Tungsten Phosphate Glasses

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**ABSTRACT:** Glass of the system: (45-x) P<sub>2</sub>O<sub>5</sub>:10ZnO:10Li<sub>2</sub>O:10PbO:25WO<sub>3</sub>:xHo<sub>2</sub>O<sub>3</sub> (where x=1, 1.5, 2 mol %) have been prepared by melt-quenching method. (where x=1, 1.5 and 2 mol%) have been prepared by melt-quenching technique. The amorphous nature of the prepared glass samples was confirmed by X-ray diffraction. Optical absorption, Excitation, fluorescence and Transmittance spectra have been recorded at room temperature for all glass samples. Judd-Ofelt intensity parameters  $\Omega_{\lambda}$  ( $\lambda=2, 4$  and  $6$ ) are evaluated from the intensities of various absorption bands of optical absorption spectra. Using these intensity parameters various radiative properties like spontaneous emission probability (A), branching ratio ( $\beta$ ), radiative life time ( $\tau_R$ ) and stimulated emission cross-section ( $\sigma_p$ ) of various emission lines have been evaluated.

**KEYWORDS:** ZLLTP Glasses, Optical Properties, Judd-Ofelt Theory, Transmittance spectra.

## I. INTRODUCTION

Glass as a material is comparatively cheap, relatively easy to produce as well as use for synthesis in a wide range of compositions. Glass is an amorphous solid with complete absence of long range order and periodicity, exhibiting a glass transition region. This amorphous material is also known as super cooled liquid due to the rapid cooling of the molten mass into the rigid state and due to lack of its first order phase transition where volume, entropy and enthalpy are discontinuous. Glass is flexible and a good host for rare-earth (RE) ions. It exhibit high optical transparency, good rare earth ion solubility, Phosphate glass has high mechanical, good chemical and high thermal stability [1-8]. Among different glass hosts, phosphate glasses have unique properties. They present superior properties that include high thermal stability, high transparency, a low melting point, a high gain density and low dispersion rates. Due to their low phonon energies, the host materials have been effectively used to increase the efficiency of the radiative emissions from the ion doped glass [9]. Phosphate glasses have been investigated as rare earth ion host matrices in laser systems, fibers and optical lenses. An important issue of this phosphate glass is low chemical stability and durability [10-13].

The present work reports on the preparation and characterization of rare earth doped heavy metal oxide (HMO) glass systems for lasing materials. I have studied on the Optical absorption, Excitation, fluorescence and Transmittance spectra properties of Ho<sup>3+</sup> doped zinc lithium lead tungsten phosphate glasses. The intensities of the transitions for the rare earth ions have been estimated successfully using the Judd-Ofelt theory, The laser parameters such as radiative probabilities (A), branching ratio ( $\beta$ ), radiative life time ( $\tau_R$ ) and stimulated emission cross section ( $\sigma_p$ ) are evaluated using J.O. intensity parameters ( $\Omega_{\lambda}$ ,  $\lambda=2, 4$  and  $6$ ).

## II. EXPERIMENTAL TECHNIQUES

### Preparation of glasses

The following Ho<sup>3+</sup> doped phosphate glass samples (45-x) P<sub>2</sub>O<sub>5</sub>:10ZnO:10Li<sub>2</sub>O:10 PbO: 25WO<sub>3</sub>: xHo<sub>2</sub>O<sub>3</sub> (where x=1, 1.5 and 2 mol%) have been prepared by melt-quenching method. Analytical reagent grade chemical used in the present study consist of P<sub>2</sub>O<sub>5</sub>, ZnO, Li<sub>2</sub>O, PbO, WO<sub>3</sub> and Ho<sub>2</sub>O<sub>3</sub>. They were thoroughly mixed by using an agate pestle mortar. then melted at 1055<sup>0</sup>C by an electrical muffle furnace for 2h., After complete melting, the melts were quickly poured in to a preheated stainless steel mould and annealed at temperature of 350<sup>0</sup>C for 2h to remove thermal strains and stresses. Every time fine powder of cerium oxide was used for polishing the samples. The glass samples so



prepared were of good optical quality and were transparent. The chemical compositions of the glasses with the name of samples are summarized in **Table 1**.

**Table 1.**

Chemical composition of the glasses

Sample	Glass composition (mol %)
ZLLTP (UD)	45P <sub>2</sub> O <sub>5</sub> :10ZnO:10Li <sub>2</sub> O:10PbO:25WO <sub>3</sub>
ZLLTP (HO1)	44 P <sub>2</sub> O <sub>5</sub> :10ZnO:10Li <sub>2</sub> O:10PbO:25WO <sub>3</sub> :1 Ho <sub>2</sub> O <sub>3</sub>
ZLLTP(HO1.5)	43.5 P <sub>2</sub> O <sub>5</sub> :10ZnO:10Li <sub>2</sub> O:10PbO:25WO <sub>3</sub> :1.5 Ho <sub>2</sub> O <sub>3</sub>
ZLLTP (HO2)	43P <sub>2</sub> O <sub>5</sub> :10ZnO:10Li <sub>2</sub> O:10PbO:25WO <sub>3</sub> : 2 Ho <sub>2</sub> O <sub>3</sub>

ZLLTP (UD) -Represents undoped Zinc Lithium Lead Tungsten Phosphate glass specimens.

ZLLTP (HO) -Represents Ho<sup>3+</sup> doped Zinc Lithium Lead Tungsten Phosphate glass specimens.

### III. THEORY

#### 3.1 Oscillator Strength

The intensity of spectral lines are expressed in terms of oscillator strengths using the relation [14].

$$f_{\text{expt.}} = 4.318 \times 10^{-9} \int \epsilon(\nu) d\nu \quad (1)$$

where,  $\epsilon(\nu)$  is molar absorption coefficient at a given energy  $\nu$  (cm<sup>-1</sup>), to be evaluated from Beer–Lambert law.

Under Gaussian Approximation, using Beer–Lambert law, the observed oscillator strengths of the absorption bands have been experimentally calculated [15], using the modified relation:

$$P_m = 4.6 \times 10^{-9} \times \frac{1}{cl} \log \frac{I_0}{I} \times \Delta\nu_{1/2} \quad (2)$$

where  $c$  is the molar concentration of the absorbing ion per unit volume,  $l$  is the optical path length,  $\log I_0/I$  is optical density and  $\Delta\nu_{1/2}$  is half band width.

#### 3.2. Judd-Ofelt Intensity Parameters

According to Judd [16] and Ofelt [17] theory, independently derived expression for the oscillator strength of the induced forced electric dipole transitions between an initial  $J$  manifold  $|4f^N(S, L) J\rangle$  level and the terminal  $J'$  manifold  $|4f^N(S', L') J'\rangle$  is given by:

$$\frac{8\pi^2 m c \nu}{3h(2J+1)n} \frac{1}{n} \left[ \frac{(n^2+2)^2}{9} \right] \times S(J, J') \quad (3)$$

Where, the line strength  $S(J, J')$  is

$$S(J, J') = e^2 \sum \Omega_\lambda \langle 4f^N(S, L) J \| U^{(\lambda)} \| 4f^N(S', L') J' \rangle^2 \quad (4)$$

$\lambda = 2, 4, 6$

In the above equation  $m$  is the mass of an electron,  $c$  is the velocity of light,  $\nu$  is the wave number of the transition,  $h$  is Planck's constant,  $n$  is the refractive index,  $J$  and  $J'$  are the total angular momentum of the initial and final level respectively,  $\Omega_\lambda$  ( $\lambda=2, 4$  and  $6$ ) are known as Judd-Ofelt intensity parameters.

#### 3.3 Radiative Properties

The  $\Omega_\lambda$  parameters obtained using the absorption spectral results have been used to predict radiative properties such as spontaneous emission probability ( $A$ ) and radiative life time ( $\tau_R$ ), and laser parameters like fluorescence branching ratio ( $\beta_R$ ) and stimulated emission cross section ( $\sigma_p$ ).

The spontaneous emission probability from initial manifold  $|4f^N(S', L') J'\rangle$  to a final manifold  $|4f^N(S, L) J\rangle$  is given by:



$$A [(S', L') J'; (S, L) J] = \frac{64 \pi^2 \nu^3}{3h(2J'+1)} \left[ \frac{n(n^2+2)^2}{9} \right] \times S(J', \bar{J}) \quad (5)$$

Where,  $S (J', J) = e^2 [\Omega_2 \| U^{(2)} \|^2 + \Omega_4 \| U^{(4)} \|^2 + \Omega_6 \| U^{(6)} \|^2]$

The fluorescence branching ratio for the transitions originating from a specific initial manifold  $| 4f^N (S', L') J' \rangle$  to a final many fold  $| 4f^N (S, L) J \rangle$  is given by

$$\beta [(S', L') J'; (S, L) J] = \sum_{S, L, J} \frac{A[(S', L) J]}{A[(S', L') J' (\bar{S}, \bar{L}) \bar{J}]} \quad (6)$$

where, the sum is over all terminal manifolds.

The radiative life time is given by

$$\tau_{rad} = \sum_{S, L, J} A[(S', L') J'; (S, L) J] = A_{Total}^{-1} \quad (7)$$

where, the sum is over all possible terminal manifolds. The stimulated emission cross-section for a transition from an initial manifold  $| 4f^N (S', L') J' \rangle$  to a final manifold  $| 4f^N (S, L) J \rangle$  is expressed as

$$\sigma_p(\lambda_p) = \left[ \frac{\lambda_p^4}{8\pi c n^2 \Delta\lambda_{eff}} \right] \times A[(S', L') J'; (\bar{S}, \bar{L}) \bar{J}] \quad (8)$$

where,  $\lambda_p$  the peak fluorescence wavelength of the emission band and  $\Delta\lambda_{eff}$  is the effective fluorescence line width.

### 3.4 Nephelauxetic Ratio ( $\beta'$ ) and Bonding Parameter ( $b^{1/2}$ )

The nature of the R-O bond is known by the Nephelauxetic Ratio ( $\beta'$ ) and Bonding Parameters ( $b^{1/2}$ ), which are computed by using following formulae [18, 19]. The Nephelauxetic Ratio is given by

$$\beta' = \frac{\nu_g}{\nu_a} \quad (9)$$

where,  $\nu_a$  and  $\nu_g$  refer to the energies of the corresponding transition in the glass and free ion, respectively. The value of bonding parameter ( $b^{1/2}$ ) is given by

$$b^{1/2} = \left[ \frac{1-\beta'}{2} \right]^{1/2} \quad (10)$$

## IV. RESULT AND DISCUSSION

### 4.1 XRD Measurement

Figure 1 presents the XRD pattern of the sample contain - P<sub>2</sub>O<sub>5</sub> which is show no sharp Bragg's peak, but only a broad diffuse hump around low angle region. This is the clear indication of amorphous nature within the resolution limit of XRD instrument.

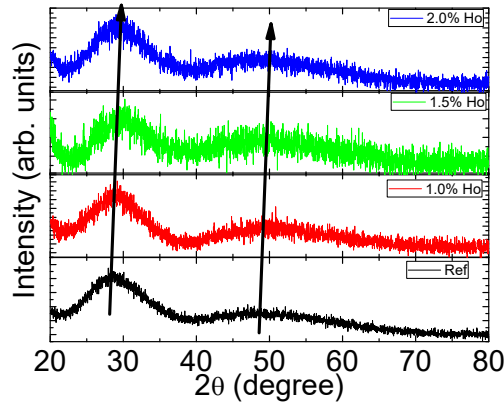


Fig. 1 X-ray diffraction pattern of  $P_2O_5:ZnO:Li_2O:PbO:WO_3:Ho_2O_3$

#### 4.2 Transmittance Spectrum

The Transmittance spectrum of  $Ho^{3+}$  doped in zinc lithium lead tungsten phosphate glass is shown in Figure 2.

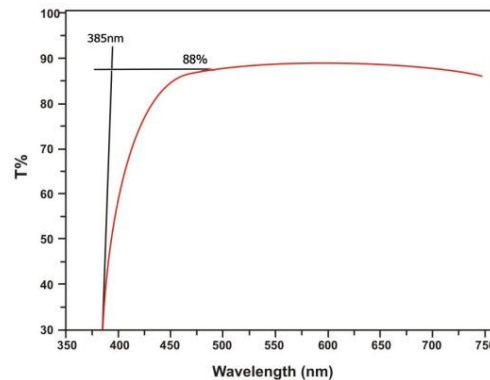


Fig. (2) Transmittance spectrum of  $Ho^{3+}$  doped ZLLTP glass.

#### 4.3 Absorption Spectrum

The absorption spectra of  $Ho^{3+}$  doped ZLLTP glass specimens have been presented in Figure 3 in terms of optical density versus wavelength. Twelve absorption bands have been observed from the ground state  $^5I_8$  to excited states  $^5I_5$ ,  $^5I_4$ ,  $^5F_5$ ,  $^5F_4$ ,  $^5F_3$ ,  $^3K_8$ ,  $^5G_6$ ,  $(^5G_6, ^3G_5)$ ,  $^5G_4$ ,  $^5G_2$ ,  $^5G_3$ , and  $^3F_4$  for  $Ho^{3+}$  doped ZLLTP glasses.

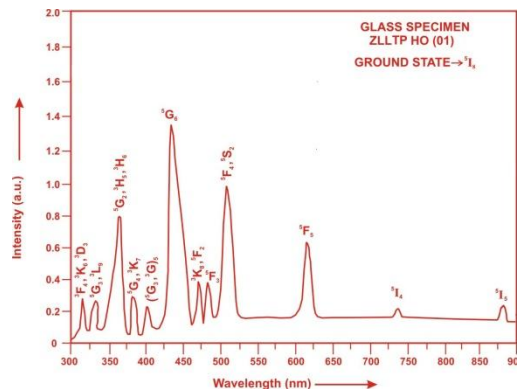


Fig. (3) Absorption spectrum of Ho<sup>3+</sup> doped ZLLTP glasses.

The experimental and calculated oscillator strength for Ho<sup>3+</sup> ions in ZLLTP glasses are given in Table 2.

Table 2: Measured and calculated oscillator strength (P<sub>m</sub> × 10<sup>+6</sup>) of Ho<sup>3+</sup> ions in ZLLTP glasses.

Energy level from <sup>5</sup> I <sub>8</sub>	Glass ZLLTP(HO01)		Glass ZLLTP(HO1.5)		Glass ZLLTP(HO02)	
	P <sub>exp.</sub>	P <sub>cal.</sub>	P <sub>exp.</sub>	P <sub>cal.</sub>	P <sub>exp.</sub>	P <sub>cal.</sub>
<sup>5</sup> I <sub>5</sub>	0.47	0.22	0.44	0.22	0.41	0.22
<sup>5</sup> I <sub>4</sub>	0.06	0.02	0.05	0.02	0.03	0.02
<sup>5</sup> F <sub>5</sub>	3.75	2.79	3.71	2.77	3.65	2.74
<sup>5</sup> F <sub>4</sub>	4.65	4.33	4.62	4.30	4.58	4.26
<sup>5</sup> F <sub>3</sub>	1.45	2.41	1.41	2.39	1.36	2.37
<sup>3</sup> K <sub>8</sub>	1.38	1.98	1.32	1.95	1.28	1.93
<sup>5</sup> G <sub>6</sub>	25.85	25.83	24.68	24.69	23.78	23.82
( <sup>5</sup> G <sub>4</sub> , <sup>3</sup> G <sub>5</sub> )	3.75	1.69	3.71	1.68	3.65	1.66
<sup>5</sup> G <sub>4</sub>	0.08	0.61	0.06	0.60	0.05	0.59
<sup>5</sup> G <sub>2</sub>	5.82	5.48	5.78	5.27	5.72	5.11
<sup>5</sup> G <sub>3</sub>	1.48	1.40	1.42	1.37	1.37	1.35
<sup>3</sup> F <sub>4</sub>	1.30	4.16	1.26	4.12	1.23	4.07
r.m.s. deviation	±1.12521		±1.12787		±1.12253	

\*Low r.m.s. deviation values clearly indicate the accuracy of fitting.

Computed values of F<sub>2</sub>, Lande's parameter (ξ<sub>4f</sub>), Nephelauxetic ratio (β') and bonding parameter (b<sup>1/2</sup>) for Ho<sup>3+</sup> ions in ZLLTP glass specimen are given in Table 3.

Table 3: F<sub>2</sub>, ξ<sub>4f</sub>, β' and b<sup>1/2</sup> parameters for Holmium doped glass specimen.

Glass Specimen	F <sub>2</sub>	ξ <sub>4f</sub>	β'	b <sup>1/2</sup>
Ho <sup>3+</sup>	358.82	1258.16	0.9337	0.1821

In the Zinc Lithium Lead Tungsten Phosphate glasses (ZLLTP)Ω<sub>2</sub>, Ω<sub>4</sub> and Ω<sub>6</sub> parameters decrease with the increase of x from 1 to 2 mol%. The order of magnitude of Judd-Ofelt intensity parameters is Ω<sub>2</sub>>Ω<sub>6</sub>>Ω<sub>4</sub> for all the glass specimens. The high values obtained for Ω<sub>2</sub> in all glasses indicate that the Ho<sup>3+</sup> ion is subjected to higher covalency with low symmetry. The spectroscopic quality factor (Ω<sub>4</sub> / Ω<sub>6</sub>) related with the rigidity of the glass system has been found to lie between 0.606 and 0.611 in the present glasses.

The values of Judd-Ofelt intensity parameters are given in Table 4.

Table 4:Judd-Ofelt intensity parameters for Ho<sup>3+</sup> doped ZLLTP glass specimens

Glass Specimens	$\Omega_2(\text{pm}^2)$	$\Omega_4(\text{pm}^2)$	$\Omega_6(\text{pm}^2)$	$\Omega_4/\Omega_6$	Ref.
ZLLTP (HO01)	6.128	1.299	2.127	0.611	[P.W.]
ZLLTP (HO1.5)	5.806	1.284	2.110	0.609	[P.W.]
ZLLTP (HO02)	5.565	1.268	2.091	0.606	[P.W.]
ZnLiBiB (HO)	4.801	0.945	1.673	0.565	[20]

#### 4.4 Excitation Spectrum

The Excitation spectra of Ho<sup>3+</sup> doped ZLLTP HO (01) glass specimen has been presented in Figure 4 in terms of Excitation Intensity versus wavelength. The excitation spectrum was recorded in the spectral region 325–525 nm fluorescence at 545nm having different excitation band centered at 347,420, 452, 474 and 485 nm are attributed to the <sup>5</sup>G<sub>3</sub>, (<sup>5</sup>G<sub>3</sub>, <sup>3</sup>G<sub>5</sub>), <sup>5</sup>G<sub>6</sub>, <sup>3</sup>K<sub>8</sub> and <sup>5</sup>F<sub>3</sub> transitions, respectively. The highest absorption level is <sup>5</sup>G<sub>6</sub> and is at 452nm. So this is to be chosen for excitation wavelength.

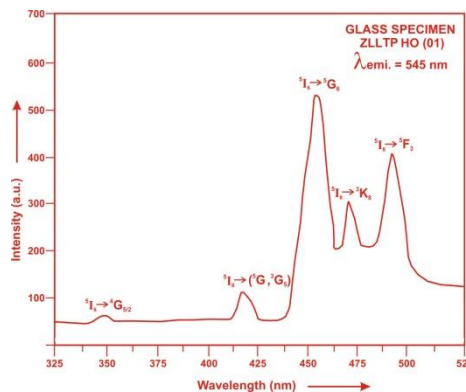


Fig. (4). Excitation spectrum of Ho<sup>3+</sup> doped ZLLTP glass.

#### 4.5 Fluorescence Spectrum

The fluorescence spectrum of Ho<sup>3+</sup> doped in zinc lithium lead tungsten phosphate glass is shown in Figure 5. There are five broad bands observed in the Fluorescence spectrum of Ho<sup>3+</sup> doped zinc lithium lead tungsten phosphate glass. The wavelengths of these bands along with their assignments are given in Table 5. The peak with maximum emission intensity appears at 555 nm and corresponds to the (<sup>3</sup>F<sub>4</sub> → <sup>5</sup>I<sub>8</sub>) transition.

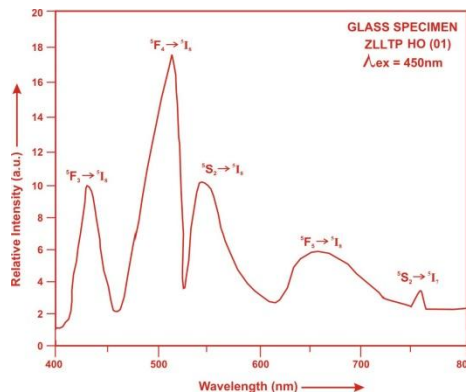


Fig. (5). Fluorescence spectrum of Ho<sup>3+</sup> doped ZLLTP glass.



**Table5:Emission peak wave lengths ( $\lambda_p$ ),radiative transition probability ( $A_{rad}$ ),branching ratio ( $\beta$ ),stimulated emission cross-section(  $\sigma_p$ ) and radiative life time(  $\tau_R$ ) for various transitions in Ho<sup>3+</sup> doped ZLLTP glasses.**

Transition	ZLLTP(HO01)					ZLLTP(HO1.5)					ZLLTP(HO02)				
	$\lambda_{max}$ (nm)	$A_{rad}(s^{-1})$	$\beta$	$\sigma_p$ ( $10^{-30} cm^2$ )	$\tau_R(\mu s)$	$A_{rad}(s^{-1})$	$\beta$	$\sigma_p$ ( $10^{-30} cm^2$ )	$\tau_R(\mu s)$	$A_{rad}(s^{-1})$	$\beta$	$\sigma_p$ ( $10^{-30} cm^2$ )	$\tau_R(10^{-20} cm^2)$		
<sup>5</sup> F <sub>3</sub> → <sup>5</sup> I <sub>8</sub>	435	4371.35	0.2651	0.6037	6063.52	4344.01	0.2651	0.5946	6101.62	4314.31	0.2653	0.5794	6150.25		
<sup>5</sup> F <sub>4</sub> → <sup>5</sup> I <sub>8</sub>	501	6938.49	0.4207	1.2537		6896.08	0.4208	1.2367		6837.32	0.4205	1.2103			
<sup>5</sup> S <sub>2</sub> → <sup>5</sup> I <sub>8</sub>	555	1822.69	0.1105	0.4378		1811.56	0.1105	0.43004		1797.93	0.1106	0.4216			
<sup>5</sup> F <sub>3</sub> → <sup>5</sup> I <sub>8</sub>	652	1975.30	0.1198	0.7428		1960.90	0.1196	0.73021		1944.31	0.1196	0.7166			
<sup>5</sup> S <sub>2</sub> → <sup>5</sup> I <sub>7</sub>	761	1384.24	0.0893	1.0182		1376.33	0.0840	1.0015		1365.64	0.0839	0.9787			

**V. CONCLUSION**

In the present study, the glass samples of composition (45-x) P<sub>2</sub>O<sub>5</sub>:10ZnO:10Li<sub>2</sub>O:10PbO:25WO<sub>3</sub>:xHo<sub>2</sub>O<sub>3</sub> (where x = 1, 1.5 and 2mol %) have been prepared by melt-quenching method. The value of stimulated emission cross-section ( $\sigma_p$ ) is found to be maximum for the transition (<sup>5</sup>F<sub>4</sub>→<sup>5</sup>I<sub>8</sub>) for glass ZLLTP (HO 01), suggesting that glass ZLLTP (HO 01) is better compared to the other two glass systems ZLLTP (HO1.5) and ZLLTP(HO02).On the basis of spectrophotometric, transmittance reaches about 88% for all phosphate glasses doped with Ho<sup>3+</sup> ions.

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